

Name I.C. LEWIS / T. PARRO  
Notebook Number 195 - 129  
Subject Chemistry of Building Materials, New Products + Experiments

Dates From \_\_\_\_\_ To \_\_\_\_\_

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52

**Subject** Initial Data of c/c Composite via BP Process  
**Cross-Reference (if any)**

(c/c comp BP)

**Purpose:**

To obtain initial wt and dimension prior to vacuum impregnation w/  
 a "T-143 TYPE" resin

**Matri:**

c/c composite via BP Process # 4-1 : Revi'd from P. S. rocky 4/30/01. From  
 1<sup>st</sup> Lawrenceburg Trial. Block 4-1 ~1.5 x 3.0 x 7.0 inches. From 4<sup>th</sup> block in  
 the series. Made w/ 0.25" long pitch fibers and kevlar 155 p. beth.  
 Wool Ratios 85/15 + 5 wt% sulfur based on the pitch weight.

**Procedure:**

Ultrasonic washed in deionized H<sub>2</sub>O 3x, 5 min intervals  $\Rightarrow$  Vacuum  
 dried overnight at  $\sim 150^{\circ}\text{C}$  to 2.4 mm pressure.

Unloaded & cooled in desiccator. 5/8/01. Weighted & dimensioned  
 5/11/01  $\Rightarrow$  loaded into oven at  $\sim 150^{\circ}\text{C}$ , 0.0 SCFH oxygen purged until  
 loaded into VI unit.

**Weight & Dimension: (5/11/01)**

wt (g)	L <sub>1</sub>	L <sub>2</sub>	L <sub>3</sub>	Ave LEN.	w <sub>1</sub>	w <sub>2</sub>	w <sub>3</sub>	Ave width	H <sub>1</sub>	H <sub>2</sub>	H <sub>3</sub>	Ave ht.	VOL. (cc)
1134.84	191.91	192.27	192.62	192.07	83.78	83.36	82.32	83.15	47.21	46.71	46.65	46.81	742.36

$$\Rightarrow \text{Density} = 1.522 \text{ g/cc}$$

**Comments:**

composite contains severe defects, such as fissures and splits. One  
 area appears to contain iron oxide.

VI and Cure: Ref. 195-124-~~555~~ 55256

Performed and Recorded by:

Directed by: J. Lin

Read and Understood by:

Date / /  
 Date / /  
 Date / /

**Subject** Preparation of ② 900 ml Aliquots of 50/50 by Volume GP-5432/Furfural (click center) 53  
**Cross-Reference (if any)**

Purpose:

To impregnate 195-129-52 w/a "T-143 Type" resin.

Materials:

GP-5432: Lot # 19588. Rec'd from Georgia-Pacific 9/28/96. LIMS # A96-03635. Stored in Freezer. Ass't Mod MCC (2) = 48.6 ( $\sigma = 0.37, n=3$ ), Brookfield Viscosity = 157.3 cPs at 71.0°F, DSC Data: Ref. 195-105-49; TGA Yield (900E) = 47.5%. Current Visc = 283.5 cPs at 68.7°F. Furfural: Reagent Grade (Fisher). Rec'd 1/15/01. 2<sup>nd</sup> Aliquot used 60 ml of ② 500 ml<sup>3</sup> rec'd 5/15/01  $\Rightarrow$  Balance = 940 ml.

Preparation 1<sup>st</sup> Aliquot: (5/14/01) - 1L Erlenmeyer Flask. 450 ml GP-5432 + 450 ml Furfural

$$\begin{aligned} \text{FLASK(etc)} + \text{GP-5432}_{\text{wt}} &= 996.3 & \text{FLASK(etc)} + \text{FURFURAL}_{\text{wt}} &= 1516.0 \\ \text{FLASK(etc)}_{\text{wt}} &= \underline{\underline{455.9}} & \text{FLASK(etc)}_{\text{wt}} &= \underline{\underline{996.3}} \\ 450 \text{ ml } \text{GP-5432}_{\text{wt}} &= 540.4 \text{ g} & 450 \text{ ml } \text{FURFURAL}_{\text{wt}} &= 519.7 \text{ g} \Rightarrow 50.98\% \text{ GP-5432 by wt} \end{aligned}$$

Comments:

May stirred w/o external heat for 10 min after combining  $\Rightarrow$  Transfer to 32 oz glass jar.

Characterization of the 1<sup>st</sup> Aliquot: (5/15/01)

Brookfield (LVT) RT Viscosity:

$$\text{Viscosity} = \underline{\underline{18.3}} \text{ cPs at } \underline{\underline{71.0}}^{\circ}\text{F} \quad \text{Spiral #1, 60 RPM, Factor = 1}$$

Spec. Grav. by RT:

$$\text{S.G.} = \underline{\underline{1.190}} \text{ at } \underline{\underline{71.8}}^{\circ}\text{F}$$

Preparation of 2<sup>nd</sup> Aliquot: (5/15/01) - 1L Erlenmeyer Flask. 450 ml GP-5432, 450 ml Furfural

$$\begin{aligned} \text{FLASK(etc)} + \text{GP-5432}_{\text{wt}} &\approx 995.0 & \text{FLASK(etc)} + \text{FURFURAL}_{\text{wt}} &= 1512.6 \\ \text{FLASK(etc)}_{\text{wt}} &= \underline{\underline{455.9}} & \text{FLASK(etc)}_{\text{wt}} &= \underline{\underline{995.0}} \\ \text{GP-5432}_{\text{wt}} &= 539.1 \text{ g} & \text{FURFURAL}_{\text{wt}} &= 517.6 \text{ g} \Rightarrow 51.02\% \text{ GP-5432 by wt} \end{aligned}$$

Solutions combined 5/16/01. Label as 195-129-53 1<sup>st</sup> use: Ref. 195-129-54

Mod MCC of Aliquot #1: Ref. 195-129-54

Performed and Recorded by:

Date

Date

Date

Directed by:

Read and Understood by:

Material:

195-129-53, 1<sup>st</sup> Aliq ref : 50/50 by volume, 51.0/49.0 by weight GP-5432/TG  
Prep. 5/14/01, Visc<sub>144</sub> = 18.3 cP at 31.6°F, SG<sub>40/4</sub> = 1.190 at 71

modMCC determinations: (5/15/01) - ~1.5g sample size. Cured 1hr at 144°F, 0.03074

$$\begin{aligned} 1) \text{CAVC} + \text{CHIPS} + \text{SAMPLE}_{(1)} &= 19.1507 \quad \text{CAVC} + \text{CHIPS} + \text{SAMPLE}_{(144)} = 18.3297 \Rightarrow \text{Yield}_{(144)} = 50.5\% \\ \text{CAVC} + \text{CHIPS}_{(1)} &= 17.6308 \quad \text{CAVC} + \text{CHIPS} + \text{SAMPLE}_{(\text{mcc})} = 18.1502 \Rightarrow \text{Yield}_{(\text{mcc})} = 67.5\% \\ \text{SAMPLE}_{(1)} &= 1.5129 \end{aligned}$$

$$\text{TOTAL YIELD} = (0.505 \times 0.6755) \times 100 = \underline{\underline{34.17\%}}$$

$$\begin{aligned} 2) \text{CAVC} + \text{CHIPS} + \text{SAMPLE}_{(1)} &= 18.9635 \quad \text{CAVC} + \text{CHIPS} + \text{SAMPLE}_{(144)} = 18.2000 \Rightarrow \text{Yield}_{(144)} = 49.20\% \\ \text{CAVC} + \text{CHIPS}_{(1)} &= 17.4606 \quad \text{CAVC} + \text{CHIPS} + \text{SAMPLE}_{(\text{mcc})} = 17.9770 \Rightarrow \text{Yield}_{(\text{mcc})} = 69.84\% \\ \text{SAMPLE}_{(1)} &= 1.5029 \end{aligned}$$

$$\text{TOTAL YIELD} = (0.4920 \times 0.6984) \times 100 = \underline{\underline{34.36\%}}$$

$$\begin{aligned} 3) \text{CAVC} + \text{CHIPS} + \text{S.4MPG}_{(1)} &= 18.3929 \quad \text{CAVC} + \text{CHIPS} + \text{SAMPLE}_{(\text{mcc})} = 17.6460 \Rightarrow \text{Yield}_{(144)} = 50.5\% \\ \text{CAVC} + \text{CHIPS}_{(1)} &= 16.8837 \quad \text{CAVC} + \text{CHIPS} + \text{SAMPLE}_{(\text{mcc})} = 17.1407 \Rightarrow \text{Yield}_{(\text{mcc})} = 68.6\% \\ \text{SAMPLE}_{(1)} &= 1.5092 \end{aligned}$$

$$\text{TOTAL YIELD} = (0.5051 \times 0.6869) \times 100 = \underline{\underline{34.69\%}}$$

Comments:

All samples were hard at temperature after cure at ~144°F.

$$\text{Ave Yield}_{(144)} = 50.1\%, \sigma = 0.78, n = 3$$

$$\text{Ave Yield}_{(\text{mcc})} = 68.7\%, \sigma = 1.15, n = 3$$

$$\text{Ave ModMCC} = 34.4\%, \sigma = 0.26, n = 3$$

Performed and Recorded by:

Directed by:

Read and Understood by:

Date:

Date:

Date:

Subject VI + Cure to 200°C of 195-129-52 #4-1 w/195-129-53  
Cross-Reference (if any)

(clc composite) 55

Purpose:

To densify clc composite w/ a phenolic resin/furfural blend. To verify max vol% pitch w/ pitches in the PSC.

Materials:

- 1) clc composite: 195-129-52 #4-1 (clc composite via AP process). From 4<sup>th</sup> Block in the 1<sup>st</sup> Lawrenceburg Trial. 0.25" long pitch fibers + Reilly 105 Pitch. Load ratio 85/15 + 5 wt% sulfur based on the pitch wt.) Wt., = 1138.84g, Vol., = 748.383cc, Dens., = 1.522g/cc
- 2) Impregnant: 195-129-53 (50/50 by volume GP-5432/Furfural, Prep. 5/14+15/01.  
 $V_{IS(2)} = (7.4) \text{ cPS at } 74.5^\circ\text{F}, S.G.(51 \pm 1.188) \text{ at } 74.5^\circ\text{F}$

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15+16

Pump-down Data: (5/14-16/01)

DATE	TIME	PRESS (mTorr)	Comments
5/14	13:30	15	load clc composite fram over (i.e. ~150°C, Atm. Pressure).
"	13:40	4	
"	16:05	16	
5/15	7:20	14	
"	16:00	16	
5/16	7:25	13	
"	8:05	15	charge traps w/ dry ice-acetone
"	8:50	10	LDR
"	11:45	10	Begin VT

Impregnation Data: (5/16+17/01) - LDR w/traps charged

LDR: Initial = 10 millitorr  $V_{IS(2)} = 17.4 \text{ cPS at } 74.5^\circ\text{F}$  Drop Time = 11:45 (10 mTorr)  
5 min = 21 "  $S.G.(51 \pm 1.188) \text{ at } 74.5^\circ\text{F}$  On-load Time = 8:45 (5/17/01)  
10 min = 27 " Held at atmospheric pressure for  
15 min = 34 " ~21 hr.

Comments:

570 ml of impregnant in the 500ml cylinder funnel.

cont'd next page

Performed and Recorded by:

Directed by: J. Caw

Read and Understood by:

Date , ,

Date

Date

**Subject**  
VI and Cure ( $\sim 150^{\circ}\text{C}$ ) Data of 195-129-52 #4-1 w/ 195-129-53  
**Cross-Reference (if any)**

(C/L COMP RT)

Post Impregnation Data: (5/17/01)

$$W_{\text{CPI-1}} = 1320.11\text{g} \Rightarrow W + \text{Pickup} = \underline{\underline{181.27\text{g}}} \Rightarrow W/\text{Pickup} = \underline{\underline{15.92}}, \text{ Vol}/\text{Pickup} = \underline{\underline{20.40}}$$

$$\text{At Pan + Screen } \underline{\underline{181.4\text{g}}}$$

Curing Data: (5/17/01) - Cure in small (1 A) Pan w/ an s.s. screen to determine amount of run-out.

TIME	EVEN SET	EVEN TEMP	Comments
* 8:45	48	158	Load into oven. Purge w/ argon at 5.0 SCFH (412)
9:15	"	156	Wet resin on all visible surfaces. Condensation on oven clear.
9:30	"	154	Impregnant boiling on surfaces. Runout on screen & in the pan.
10:05	"	158	Boiling has ceased. Resin is likely cured.
- (2) 10:40	"	156	Unload to desiccator. Set oven at $\sim 82^{\circ}\text{C}$ (81%). Cool s/s components & weights.
-	-	-	$W_t = 1232.35\text{g} \Rightarrow W + \text{Pickup} = 93.51\text{g} \Rightarrow W/\text{Pickup} = 8.21$ ( $\text{Yd} = \underline{\underline{51.6\%}}$ )
-	-	-	$\text{Pan + Screen + Resid} = 188.4\text{g} \Rightarrow \text{Cured Run-out} = \underline{\underline{7.0\text{g}}}$
* 11:55	82	245	Load over (180° Rotation $\rightarrow$ to bottom).
- (3) 13:55	"	248	Power off. Allow oven to cool to $\sim 150^{\circ}\text{C}$ $\Rightarrow$ unload to desiccator
15:25	OFF	156	Unload to desiccator. Cool overnight $\Rightarrow$ weigh following morning

Post Curing Data: (5/18/01)

$$\text{Pan + Screen + Cured Run-out}_{(P)} = 188.3\text{g} \Rightarrow \text{Cured Run-out} = \underline{\underline{6.9\text{g}}}$$

$$W_{\text{DM}} = 1212.74\text{g} \Rightarrow W + \text{Pickup} = 73.2\text{g} \Rightarrow W/\text{Pickup} = \underline{\underline{6.43}} \quad (\text{Yd} = 40.8\%)$$

$$\Rightarrow \text{Impregnate Yield (including the run-out)} = \underline{\underline{44.6\%}} \Rightarrow \text{Density}_{\text{LS}} \approx \underline{\underline{1.620 \text{ g/cc}}}$$

Label 195-129-56, Give to P. S. Rocky, 5/21/01

Rebunker Factor: R.F. 145-129-76

Performed and Recorded by:

Directed by: J. C. Liu

Read and Understood by:

Date: 1/1

Date: ...

Date: ..

Material:

195-129-56: c/c composite via BP process from the 4<sup>th</sup> Block in the 1<sup>st</sup> Lawrenceburg trial. 0.25" long pitch fibers + Reilly 155 pitch. Load Factor 85/15 + 5 wt% sulfur based on pitch wt. Initial Data: Ref. 195-129-52 #4-1. VI & CURE Data: Ref. 195-129-55 & 56. Rec'd from P. Sirocky 6/20/01.

Rebake Cycle (per P. Sirocky):

10°C/hr to 900°C, 2 hr hold. Block was warm when received. Cooled in desiccator  $\Rightarrow$  weighed & dimensioned.

Rebake Data: (6/20/01)

WT (g)	L <sub>1</sub>	L <sub>2</sub>	L <sub>3</sub>	AVE. LEN.	W <sub>1</sub>	W <sub>2</sub>	W <sub>3</sub>	AVE WIDTH	H <sub>1</sub>	H <sub>2</sub>	H <sub>3</sub>	Ave HT.	Vol (cc)
1129.43	192.10	192.32	192.37	192.36	83.98	83.62	82.86	83.49	46.55	46.42	46.60	46.59	742.836

Rebake Dens. by = 1.577 g/cc  $\Rightarrow \Delta = 0.055$  g/cc over "green" density.

Wt% (green thru rebake) = 3.56%

Vol% (green thru rebake) = -0.07% (essentially no change)

Delta (green thru rebake) = 3.61%.

Wt% (cure thru rebake) = -2.75%.

Returned to P. Sirocky 6/20/01. Data communicated via e-mail to D. Hoang, P. Sirocky, & E. Paquet.

Comments:

\* Impregnant % wt Yield from Vf thru Rebake =  $(40.59 / 181.27) \times 100 = 22.4\%$ .

Note:

z. Mod MCC = 34.4 ( $r=0.26, n=3$ ) for the impregnant  $\Rightarrow$  The difference is due to run-out and/or weight loss from the pitch binder because it hadn't been to ~900°C yet.

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

**Subject** Initial Data of C/C Composites via BP Process (3<sup>rd</sup> Trial) (C/C Comp)  
**Cross-Reference (if any)**

Purpose:

To obtain initial weights and dimensions prior to vacuum impregnation with silicon (IV) oxide colloidal dispersion for ins. to conversion to SIC.

Materials:

C/C composite via BP process. Rec'd from P. Strokey 7/16/01. Two brick sections in ~ half. from the 3<sup>rd</sup> Lawrenceburg trial. Made w/ 0.25" long K-223SE pitch fibers and Railey 155 pitch. Load Ratio: 75/25 w/o sulfur.

Procedure:

Ultrasonic washed in deionized H<sub>2</sub>O 3x, 5 min intervals.  $\Rightarrow$  Vacuum dried on at ~166 °C, 0.1 mm pressure.

Air cooled, will not fit in desiccator,  $\Rightarrow$  weighed & dimensioned.

Initial Data: (7/19/01)

File Path = c:\Program Files\Excel\BP C-C Composites\Initial.xls Sheet = BP III

**BP C/C COMPOSITES INITIAL WEIGHTS AND DIMENSIONS**

Material:

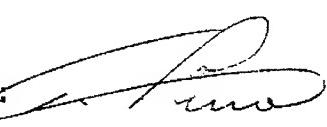
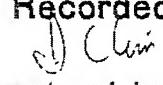
Material: BP-III-1 and BP-III-2. Rec'd 7/16/01. Ultrasonic washed 3x for 5 min. in deionized water on 7/19/01. Dimensions were obtained with a Starrett No. 123-12 vernier caliper. Not vacuum dried at 166 °C to 0.1 mm pressure from 7/18 to 7/19/01. Weights obtained on Mettler PN 2210 balance on 7/19/01.

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Dens. (g/cc)
3-1-A	916.19	8.173	8.155	8.110	8.146	3.261	3.305	3.304	3.290	1.333	1.302	1.288	1.308	574.297	1.595
3-1-B	823.65	8.196	8.200	8.197	8.198	3.296	3.271	3.248	3.272	1.253	1.273	1.259	1.262	554.502	1.485
<b>AVERAGE =</b>															
<b>STND. DEV. =</b>															

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Dens. (g/cc)
3-2-A	867.13	8.883	8.863	8.837	8.861	3.226	3.265	3.298	3.263	1.183	1.170	1.169	1.174	556.247	1.559
3-2-B	861.58	8.913	8.905	8.896	8.905	3.263	3.281	3.292	3.279	1.195	1.207	1.180	1.194	571.400	1.508
<b>AVERAGE =</b>															
<b>STND. DEV. =</b>															

Dimensioned: 07/19/01  
 Not Vac. Dried: 07/18-19/01  
 Weighed: 07/19/01  
 N.B. Ref. No.: 195-129-86

#	Impregnant	NB Reference
3-1-A	Silicon (IV) Oxide Colloidal Dispersion	195-129-88+84
3-1-B	" " "	195-129-92+93
3-2-A	" " "	2VJ 195-129-94+95
3-2-B	" " "	195-129-96+97

Performed and Recorded by:   
 Directed by:   
 Read and Understood by:

Date:  
 Date:  
 Date:

**Subject:** VI of 195-129-86 #3-1-A w/ SICer (IV) Oxide Colloidal Dispersion C/c com  
**Cross-Reference (if any)**

Purpose:

To investigate insitu conversion of carbon to SiC in a c/c composite

Materials:

- 1) c/c Composite: 195-129-86 #3-1-A; (c/c composite via BP process from the 1<sup>st</sup> Bl of the 3<sup>rd</sup> Lawrenceburg trial. 0.25" long K-223SE pitch Fibers and Reiley 153 pi. Load ratio 75/25. No sulfur. Wt<sub>c/c</sub> = 916.19 g, Vol<sub>c/c</sub> = 574.297 cc, Dens<sub>c/c</sub> = 1.595 g
- 2) Impregnant: Silicon (IV) Oxide, 30% in H<sub>2</sub>O, colloidal dispersion. (Alfa-Aesar), lot # A041309, 0.01 um particles, in liquid, S.A. = 320 m<sup>2</sup>/g. Density = Visc<sub>c/c</sub> = 7.7 cps at 82.1°F, S.G.<sub>c/c</sub> = 1.216 at 82.1°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15 & 16 Use teflon support and Pyrex pan for drying.

Pump Down Data: (7/20-23/01)

DATE	TIME	PRESS (inert)	Comments
7/20	12:45	18	Load c/c composite from cold, dry oven.
"	13:00	"	Begin pump-down.
"	14:00	44	
"	15:05	28	
7/23	7:20	21	
"	8:00	"	Charge traps w/ dry ice-acetone.
"	9:05	14	LDR
"	11:30	13	Begin VI

Impregnation Data: (7/23 & 24/01) - LDR w/traps charged

LDR: Init. Pres = 14 mTorr Visc<sub>c/c</sub> = 7.7 cps at 82.1°F  
 5 min = 27 " S.G.<sub>c/c</sub> = 1.216 at 82.1°F  
 10 min = 38 "  
 15 min = 49 "

Drop time = 11:50 (13 mTorr)

Unload time = 8:00 (7/24)

Held at atmospheric pres.  
 for ~20 1/4 hrs.

Comments:

500 cc cylindrical funnel is full. Return impregnant to ② lqt. poly-bet

Post Impregnation Data: (7/24/01)

$$Wt_{(PVI.1)} = 1029.86 \text{ g} \Rightarrow Wt_{P.2cup} = 113.67 \text{ g} \Rightarrow Wt_{1/2 Pickup} = 12.41 \text{ g} \quad \text{Vol}_{1/2 Pickup} = 46.0 \text{ ml}$$

Performed and Recorded by: J. L. Lin

Date: \_\_\_\_\_

Directed by: J. L. Lin

Date: \_\_\_\_\_

Read and Understood by:

Date: \_\_\_\_\_

**Subject** VI-195-129-86 #3-1-A wtsil. crystall. & Colloidal dispersion (c/c compn) 89  
**Cross-Reference (if any)**

Drying Data: (7/24/25/01) - oven set "29" (50%). Argon purge = 5.0 SCFH (A/R)

TIME	OVEN TEMP	PRESS (mm)	Comments
* 8:10	108	Atm	Load oven; # to top. Oven set "29" (50%). Purge w/argon at 5.0 SCFH (A/R)
(3) 11:10	106	"	Unload oven. Set oven at "38" (50%). Weigh brick hot (it doesn't fit in balance)
-	-	-	Wt = 989.02g $\Rightarrow$ Wt. Pickup = 72.81g $\Rightarrow$ Wt/o Pickup = <u>7.95</u> (Yd = <u>64.1%</u> )
* 11:30	125	Atm	Load oven; # to bottom. Oven set at "38" (50%). Purge w/argon at 5.0 SCFH (A/R)
(2) 13:30	130	"	Unload oven. Set oven at "46" (50%). Weigh brick hot. Install new gasketing.
-	-	-	Wt = 954.41g $\Rightarrow$ Wt. Pickup = 38.22g $\Rightarrow$ Wt/o Pickup = <u>4.17</u> (Yd = <u>33.4%</u> )
* 14:20	126 (7)	736.6	Load oven; # to top. Oven set "46" (50%). Vac. pump on. Argon purge off. Reduce pressure.
(7) 7:20	170	0.4	Vac. pump off. Pressure rise w/argon. Set oven at "29" (50%). Leave door open to cool.
-	-	-	Wt = 952.26g $\Rightarrow$ Wt. Pickup = 34.07g $\Rightarrow$ Wt/o Pickup = <u>3.94</u> (Yd = <u>31.7%</u> )

Comments:

1) After 3 hrs,  $\sim 107^\circ\text{C}$ , atm pressure;

No evidence of run-out. Set oven at "38" (50%). Rotate brick 180° (# to bottom).

2) After 2 hrs,  $\sim 128^\circ\text{C}$ , atm pressure;

No evidence of run-out. Set oven at "46" (50%). Rotate brick 180° (# to top). Remove old door gasketing and replace w/new gasketing

3) After 17 hrs,  $\sim 175^\circ\text{C}$ , vacuum

Note: by 15:30 (7/24/01) the oven temperature was  $\sim 160^\circ\text{C}$  and the pressure was 0.01mm  $\Rightarrow$  sample dried quickly.

No evidence of run-out. Set oven to "29" (50%) and leave door open to cool.

Comments:

The percent weight yield of the impregnant, *in situ*, agrees w/that of the solution in crucible. Ref. 195-129-90.

Label 195-129-89

Performed and Recorded by:

Directed by: J. Caw

Read and Understood by:

Date - / /

Date

Date

**Subject** Drying of ~10g samples of silicon (IV) Oxide, 30% in H<sub>2</sub>O, colloidal dispersion (decomp)  
**Cross-Reference (if any)**

Purpose:

To determine the percent yield by weight of solution to compare w/ insitu percent weight yield of Vfcl c/c composites.

Material:

silicon (IV) oxide, 30% in H<sub>2</sub>O, colloidal dispersion (AlF<sub>3</sub>-Aesop), 40+<sup>#</sup> A04K09,  
0.01 um particles, in liquid. SA = 320 m<sup>2</sup>/g. Density = 1.20.  
 $V_{B(C)} = \frac{7}{12} \text{ cps at } 82.1^{\circ}\text{F}$ , Spec. Grav.<sub>r1</sub> = 1.216 at 82.1 °F.

Apparatus:

② 100 ml porcelain. Al foil covers.

Procedure:

Weigh ~10g into each crucible, containing SiC boiling chips. Cover w/ Al foil.  
 Punch holes in Al foil and obtain total weight. Subtract sample weight  
 to obtain TARE. Processed w/ 195-129-89.

Initial Data: (7/24/01)

$$\begin{aligned} 1) \text{CRUC CHIPS + SAMPLE (I)} &= 59.4475 (\dagger) \quad \text{CRUC (etc) + Al Foil}_{(I)} = 60.1600 \\ \text{CRUC + CHIPS}_{(I)} &= \underline{49.3229} \quad \text{CRUC (etc)}_{(I)} = \underline{59.4475} \\ \text{SAMPLE}_{(I)} &= \underline{10.1746g} \quad \text{Al Foil}_{(I)} = \underline{0.6620g} \Rightarrow \text{TARE} = \underline{49.4854}. \end{aligned}$$

$$\begin{aligned} 2) \text{CRUC + CHIPS + SAMPLE (II)} &= 54.7967 (\dagger) \quad \text{CRUC (etc) + Al Foil}_{(II)} = 55.4580 \\ \text{CRUC + CHIPS}_{(II)} &= \underline{44.7620} \quad \text{CRUC (etc)}_{(II)} = \underline{54.7967} \\ \text{SAMPLE}_{(II)} &= \underline{10.0347g} \quad \text{Al Foil}_{(II)} = \underline{0.6613g} \Rightarrow \text{TARE} = \underline{45.4233}. \end{aligned}$$

Drying Data: (7/24+25/01) - Processed w/ 195-129-89

	TIME	OVEN TEMP	PRES (mm)	Comments
*	8:10	108	Atm	Load in oven; in front of Pyrex tray w/ 195-129-89. Purge w/larger at 5.0 scFH(Gas)
(3)	11:10	106	"	Unload, cool in desiccator $\Rightarrow$ weigh. Set over at "38" (50%).
-	-	-	"	#1 Cruc (etc) Wt = 54.7662g $\Rightarrow$ Wt = 4.7808g $\Rightarrow$ Wt Yield = 46.99%
-	-	-	"	#2 Cruc (etc) Wt = 49.9430g $\Rightarrow$ Wt = 4.5197g $\Rightarrow$ Wt Yield = 45.04%
*	11:30	125	Atm	Reload; switch sides. Over set "38" (50%). Purge w/larger at 5.0 scFH(Gas)
(2)	13:30	130	"	Unload, cool in desiccator $\Rightarrow$ weigh. Set over at "46" (50%).
-	-	-	"	#1 Cruc (etc) Wt = 53.2721g $\Rightarrow$ Wt = 3.2867g $\Rightarrow$ Wt Yield = 32.30%.
-	-	-	"	#2 Cruc (etc) Wt = 48.6710g $\Rightarrow$ Wt = 3.2477g $\Rightarrow$ Wt Yield = 32.36%
				cont'd next page

Performed and Recorded by: J. Lew

Date

Directed by: J. Lew

Date

Read and Understood by:

Date

**Subject** Drying of ~10g samples of S.I. iron (II) Oxide, 3wt% in H<sub>2</sub>O, colloidal dispersion (etc contd BPI) 91  
**Cross-Reference (if any)**

Drying Data (cont'd): (7/24 + 25/61)

TIME	OVEN TEMP (°F)	PRESS (mm)	Comments
* 14:20	126 (n)	736.6	Reload; reverse sides. Or use set "40" OVEN. Vac. pump off. Argon purge off.
• 17:20	175	0.4	Vac. pump off. Pressurize stronger. Unload, cool in desiccator. → weigh.
-	-	-	① (calc'd) $W_t = 53.2182g \Rightarrow W_t = 3.2328g \Rightarrow W_t Y_{rel} = 31.77\%$
-	-	-	② (calc'd) $W_t = 48.6127g \Rightarrow W_t = 3.1894g \Rightarrow W_t Y_{rel} = 31.78\%$

Ave % wt yield = 31.8,  $\sigma = 0.01$ ,  $n = 2$

Comments:

Almost exactly the same value for each sample. Same percent weight yield as the insitu 1. wt.yield for 195-127-89.

Performed and Recorded by:

Directed by: *J. Lin*

Read and Understood by:

Date

Date

Date

**Subject:** VI-F 195-129-86 #3-1-B w/ silicon (IV) oxide, Colloidal Dispersion (c/c com  
Cross-Reference (if any)

Purpose:

Ref. 195-129-88

Materials:

- 1) c/c Composite: 195-129-86 #3-1-B; c/c composite via BP process from the 1<sup>st</sup> block of the 3<sup>rd</sup> Lawrenceburg trial. 0.25" long K-223-SE pitch fibers and Reiley 105, load ratio 75/25. No sulfur. Wt (2) = 823.65g, Vol (2) = 554.502cc, Dens (2) = 1.485g/l
- 2) Impregnant: silicon (IV) oxide, 30% in H<sub>2</sub>O, colloidal dispersion (AIFa-Ascar), lot # A04K09 0.01 um particles, in liquid. SA = 320 m<sup>2</sup>/g. Dens. (2) = 1.20. Previous use is 195-129-8 V<sub>1.3</sub>(2) = 7.7 cps at 82.1°F, S.G.(2) = 1.216 at 82.1°F.

Apparatus:

Ref. 195-120-105

Procedure:

Ref. 195-129-88

Pump-Down Data:

DATE	TIME	PRESS (mTorr)	Comments
7/23	13:15	21	Load block from hot oven (~110°C, atm)
"	13:25	"	Begin pump-down
"	14:25	45	
7/24	7:10	24	
"	8:25	23	charge traps w/dry ice-acetone
"	8:55	15	LDR
"	11:45	13	Begin VE

Impregnation Data: (7/23+24/01) - LDR w/traps charged.

LDR: Initial = 84 mTorr      V<sub>1.3</sub> = 7.8 cps at 78.0°F      Drop Time = 11:45 - (13 mTorr)  
 5 min = 27      "      S.G. = (1.216) at 78.0°F      Unload Time = 7:45 (7/25/01)  
 10 min = 38      "      Held at atmospheric pressure for  
 15 min = 49      "      ~20 hrs.

Comments:

500 ml cylindrical funnel filled (ie - ~675 ml)

Post Impregnation Data: (7/25/01)

$$Wt_{(PV1-1)} = 950.80g \Rightarrow Wt_{\text{Pickup}} = 127.15g \Rightarrow Wt_{\text{to Pickup}} = (15.44) \text{ Vol to Pickup} = 18.86$$

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

Subject V.F. of 195-129-86 #3-1-B w/ Si<sub>3</sub>N<sub>4</sub> (II) oxide, Colloidal Dispersion (o/c comp BP) 93  
 Cross-Reference (if any)

Drying Data: (7/25 + 26/01) - over set "29" (50°C). Purge whager at 5.0 SCFH (4°C)

TIME	OVEN TEMP (m/m)	PRESS (mm)	Comments
* 8:00	107	Atm	Load oven. # to top. Over set "29" (50°C). Argon purge 5.0 SCFH (4°C)
(3) 11:00	108	"	Unload oven. Over set "38" (50°C). Weigh block hot.
-	-	-	Wt = 907.62 g $\Rightarrow$ Wt. Pickup = 83.97 g $\Rightarrow$ Wt/o Pickup = <u>10.19</u> ( $Y_d = \underline{\underline{66.0\%}}$ )
* 11:20	129	Atm	Load oven; # to bottom. Over set "38" (50°C). Argon purge 5.0 SCFH (4°C)
(2) 13:20	132	"	Unload oven. Over set "46" (50°C). Weigh block hot. Install new gasketing.
-	-	-	Wt = 871.76 g $\Rightarrow$ Wt Pickup = 48.11 g $\Rightarrow$ Wt/o Pickup = <u>5.84</u> ( $Y_d = \underline{\underline{37.8\%}}$ )
* 13:40	136 (n)	736.9	Load oven; # to top. Over set "46" (50°C). Vac. pump on. Argon purge off.
(8) 7:40	168	0.4	Vac. pump off. Presurize whager. Set oven at "29" (50°C).
-	-	-	Wt = 863.89 g $\Rightarrow$ Wt Pickup = 40.34 g $\Rightarrow$ Wt/o Pickup = <u>4.89</u> ( $Y_d = \underline{\underline{31.6\%}}$ )

Comments:

1) After 3 hrs, ~108°C, atm. pressure;  
 Rotate brick 180° (# to bottom). Set oven at "38" (50°C). No evidence of run-out.  
 Similar % wet yield to 195-129-89 (i.e. 64.1%).

2) After 2 hrs, ~131°C, atm. pressure;  
 Scrape off old gasketing and install new. Set oven to "46" (50°C). Rotate  
 brick 180° (# to top). No evidence of run-out. H<sub>2</sub>O condensed on oven door;  
 wiped dry.

3) After 18 hrs, ~168°C, vacuum;  
 No evidence of run-out. Over set to "29" (50°C). Leave door open to cool.

Label 195-129-93

Graph Data: Ref. 195-130-63

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

94

**Subject:** ~~15 VI of 195-129-86 #3-A w/ silicon (II) oxide, colloidal dispersion~~ (clc com.  
**Cross-Reference (if any)**

**Purpose:**

Ref. 195-129-88

V

**Materials:**

- 1) clc composite 195-125-86 #3-2-4 clc composite via BP process. Fiber block #2 of the 3<sup>rd</sup> Lawrenceburg trial. 0.25" long K-223SE pitch fibers. Reitley 105 pitch. Load Ratio = 7.5/1 w/o sulfur.  $W_{clc} = 867.13g$ ,  $V_{clc} = 556.247cc$ , Density =  $1.557g/cc$
- 2) Impregnant: silicon (II) oxide, 30% in  $H_2O$ , colloidal dispersion, (Alfa-Aesar), Lot # A0404. 0.01um particles, in liquid. SA =  $320m^2/g$ , Density = 1.20, Previous use: 7/25/01.  $V_{30\%} = 7.7 \text{ cps at } 82.1^\circ F$ ,  $S.G_{30\%} = 1.216 \text{ at } 82.1^\circ F$ .

**Apparatus:**

Ref. 195-120-15

**Procedure:**

Ref. 195-129-15 + 16 \* Processed w/ 195-129-86 #3-2-B

**Pump Down Data: (7/24+25/01)**

DATE	TIME	PRESS (mm)	Comments
7/24	13:20	19	Load from open lab
"	13:35	"	Begin pump-down
"	14:35	340	"High" pressure $\Rightarrow$ brittle picked up moisture in lab
"	15:55	~205	
7/25	7:20	37	
"	8:35	33(4)	Charge traps w/ dry ice-acetone
"	9:20	22	LDR
"	11:45	18	Reopen VT

**Impregnation Data: (7/25+26/01) - LDR off/traps charged**

LDR: Initial = 22 mTorr  $V_{rec} = 7.7 \text{ cps at } 78.0^\circ F$  Drop Time = 11:45 116 mTorr  
 $t_{min} = 46$  "  $S.G_{30\%} = 1.220 \text{ at } 78.0^\circ F$  U:load time = 8:15 (7/26/01)  
 $10min = 60$  " Held at atmospheric pressure  
 $15min = 74$  " for  $\sim 20\frac{1}{2}$  hrs.

**Comments:**

Effect second by third reel (future).

**Post-Impregnation Data: (7/26/01)**

$$W_{t(PV2-1)} = 987.35g \Rightarrow W_{t Pickup} = 120.22g \Rightarrow W_{t Pickup} = 13.86, V_{t Pickup} = 17$$

**Performed and Recorded by:** J. J. [Signature]

Date

Directed by: J. J. [Signature]

Date

Read and Understood by:

Date

Subject: LVI of 195-129-86 #3-2-A w/ 5.1ccm CII on dry, cold, clear/clarified dispersion (ct. compare) 95  
 Cross-Reference (if any)

Drying Data: (7/26 + 27/61) - Over set "29" (50%). Purge w/ argon at 5.0 SCFH/LAIR

TIME	OVEN TEMP	PRESS (mm)	Comments
* 8:30	106	Atm	Load oven; # to top. over set "29" (50%). Argon purge at 5.0 SCFH/LAIR
(1) 11:30	120	"	Unload oven. Over set "38" (50%). Weigh block hot.
-	-	-	Wt = 944.48g $\Rightarrow$ Wt Pickup = 77.35g $\Rightarrow$ Wt/o Pickup = 8.92 ( $Y_d = 64.3\%$ )
* 11:45	130	Atm	Load oven; # to bottom, front of tray. Over set "38" (50%). Argon purge 5.0 SCFH
(2) 13:45	138	"	Unload oven. Over set "46" (50%). Weigh block hot. Install new gasketing.
-	-	-	Wt = 906.75g $\Rightarrow$ Wt Pickup = 39.62g $\Rightarrow$ Wt/o Pickup = 4.57 ( $Y_d = 33.0\%$ )
* 14:30	176	742.0	Load oven; # to top, back of tray. Vac. pump off. Argon purge off. Reduce pressure.
(18) 8:30	166	0.3	Vac. pump off. Pressurize w/ argon. Set oven at "29" (50%).
-	-	-	Wt = 905.02g $\Rightarrow$ Wt Pickup = 37.89g $\Rightarrow$ Wt/o Pickup = 4.37 ( $Y_d = 31.5\%$ )

Comments:

1) After 3 hrs, ~113°C, atm. pressure;  
 No evidence of run-out. Set oven at "38" (50%). Weigh block hot. Rotate 180°;  
 # to bottom. Reverse position w/ 195-129-86 #3-2-B (i.e. Front of Pyrex tray).

2) After 2 hrs, ~148°C, atm pressure;  
 No evidence of run-out. Set oven at "46" (50%). Weigh block hot. Rotate 180°; # to top. Reverse position w/ "B" (i.e. rear of Pyrex tray). Remove old gasketing and install new.

3) After 18 hrs, ~171°C, vacuum)  
 Set oven at "29" (50%). Weigh block hot  $\Rightarrow$  reload w/ "B" comparison into VI unit for 2<sup>nd</sup> impregnation

Comments:

3. yield of the impregnant agrees w/ previous 2 blocks; 31.7%,  
 31.6%, and 31.5%.

Label 195-129-95 #3-2-A  $\xrightarrow{29}$  VI: Ref. 195-129-99+100

Performed and Recorded by:

Directed by: J. Clin

Read and Understood by:

Date: / /

Date: / /

Date: / /

**Subject:** VI-F 195-129-86 #3-2-B wts. heat (II) oxide, colloidal dispersion (etc comp.)  
**Cross-Reference (if any)**

Purpose:

Ref. 195-129-88

Materials:

(1) c/c composite 195-129-86 #3-2-B (c/c composite via B/P process. From block #2 of the 3rd Lawrenceburg trial, 0.25" long K-223 SE pitch fibers. Rat/leg 15% p. etc. Load ratio = 75/12 wt. wt. wt. - 861.58g, Vol. 21 = 571.400cc, Dens. = 1.508g/cc

(2) Impregnant: Silicon (II) oxide, 30% in H<sub>2</sub>O, colloidal dispersion. (A) Fm-Aesa-7400.0 plus particles, in liquid. SA = 320 m<sup>2</sup>/g. Density = 1.20. Previous use: 7/26/01. Viscosity = 7.7 cps at 82.1°F, S.G. 21 = 1.216 at 82.1°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15+16 \* Processed w/ 195-129-86 #3-2-A

Pump-down Data: (7/24/01)

DATE	TIME	PRESS (in Torr)	Comments
7/24	13:20	19	Load from open lab w/ 3-2-A
"	13:35	"	Begin pump-down.
"	14:35	370	" High pressure → blocks picked up moisture in lab.
"	15:55	205	"
7/25	7:20	370	.
"	8:35	33(0)	Charge traps w/ dry ice-acetone
"	9:20	22	LDR
"	11:45	18	Begin VI

Impregnation Data: (7/25+26/01) - LDR w/ traps charged

LDR: Initial = 22 mTorr       $\text{Visc. } 7.4 \text{ cps at } 78.0^{\circ}\text{F}$       Drop Time = 11:45 (18 mTorr)  
 $J_{\text{min}} = 46$  "       $3G_{\text{21}} + (1.226) \text{ at } 78.0^{\circ}\text{F}$       Onload Time = 8:15 (7/26/01)  
 $I_{\text{min}} = 60$  "      "      "  
 $D_{\text{min}} = 74$  "

Comments:

Filled 500ml cylindrical funnel (i.e. uozmld).

Post Impregnation Data: (7/26/01)

Wt (PyI-1) = 995.39g → Wt Pickup = 133.91g ⇒ w/o Pickup = 15.53, Vol/o Pickup = 18.20

Performed and Recorded by: J. ...

Date:

Directed by: J. ...

Date:

Read and Understood by:

Date:

Subject VI of 195-129-86 #3-2-B. 0.5% v/v (III) oxidized colloidal dispersion (click on PBA)  
 Cross-Reference (if any)

Day 25 Data: (7/26 + 27/61) - Over set "29" (50%). Argon purge 5.0 SCFH (41R). Process w/195-129-95

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:30	106	Atm	Load oven; * to top. Over set "29" (50%). Argon purge at 5.0 SCFH (41R).
(3)	11:30	120	"	Unload oven. Set oven "38" (50%). Weigh block hot.
-	-	-		Wt = 950.91g $\Rightarrow$ Wt Pickup = 89.33g $\Rightarrow$ Wt/o Pickup = 10.37 (Yd = 66.8%)
*	11:45	150	Atm	Load oven; * to bottom, back of tray. Over set "38" (50%). Argon purge 5.0 SCFH
(2)	13:45	138	"	Unload oven. Set oven "46" (50%). Weigh block hot. Install new gasketing.
-	-	-		Wt = 906.48g $\Rightarrow$ Wt Pickup = 44.90g $\Rightarrow$ Wt/o Pickup = 5.21 (Yd = 33.6%)
*	14:30	176	742.0	Load oven; * to top, front of tray. Over set "46" (50%). Vac. pump on. Argon purge off.
(6)	8:30	166	0.3	Vac. pump off. Pressureize w/argon. Set oven "29" (50%).
-	-	-		Wt = 903.59g $\Rightarrow$ Wt Pickup = 43.01g $\Rightarrow$ Wt/o Pickup = 4.86 (Yd = 31.4%)

Comments:

1) After 3 hrs, ~1/3°C, atm pressure;

No evidence of run-out. Set oven at "38" (50%). Weigh block hot. Rotate 180°;  
 \* to bottom. Reverse position in Pyrex tray w/195-129-86 #3-2-A (i.e. back of tray).

2) After 2 hrs, ~144°C, atm pressure;

No evidence of run-out. Set oven at "46" (50%). Weigh block hot. Rotate 180° \* to  
 top. Reverse position in Pyrex tray (i.e. front of tray). Remove old door gasketing  
 and install new.

3) After 18 hrs, ~171°C, vacuum;

Set oven "29" (50%). Weigh block hot  $\Rightarrow$  release w/195-129-95 #3-2-A into VI  
 unit for 2nd impregnation.

Comments:

1. yield of impregnant agrees w/ previous ③ blocks; 31.7%, 31.6%,  
 and 31.5%.

Label 195-129-97 #3-2-B      2<sup>nd</sup> VI: Ref. 195-130-01202

Performed and Recorded by:

Directed by: J. C. Am

Read and Understood by:

Date 5/11/

Date

Date

**Subject** Initial Data of Graphitized c/c Composites via BP Process (4th Trial) (6k  
Cross-Reference (if any)

Purpose:

To obtain the initial weights and dimensions prior to vacuum impregnation with "T-143" type phenolic/furfural resin blend for densification.

Materials:

c/c composites via BP process. Rec'd from P. Sirocky 7/27/01. Two sections, both graphitized. Section "A-1" had one pitch impregnation, section "B-1" did not have a PI. Both graphitized to ~3000°C

Made w/ K-2235E 0.25" long fibers and Reillyg 155 pitch. Load Ratio = 7.5/2 w/o softwr. Brick 13 of 4th trial.

Procedure:

Essentially same as 195-129-86. Except coated in desiccator

Initial Data! (7/27/01)

File Path = C:\Program Files\Excel\BP C/C Composites\In.Trial.xls Sheet = BP II

**BP C/C COMPOSITES INITIAL WEIGHTS AND DIMENSIONS**

Material:

Material: BP-IV-13 A1 and BP-IV-13 B1. Rec'd 7/25/01. Ultrasonic washed 3x for 5 min. in deionized water on 7/26/01. Dimensions were obtained with a Mitutoyo Model CD-8'CS digital caliper. Hot vacuum dried at 124 °C to 0.4 mm pressure from 7/26 to 7/27/01. Weights obtained on Mettler PN 2210 balance on 7/27/01.

Note: Both samples have been graphitized to ~3000°C. A1 has one PI. B1 has no PI.

Sample I.D.	Weight (g)	L1 (mm)	L2 (mm)	L3 (mm)	Ave. Length (mm)	W1 (mm)	W2 (mm)	W3 (mm)	Ave. Width (mm)	H1 (mm)	H2 (mm)	H3 (mm)	Ave. Height (mm)	Vol. (cc)	Dens. (g/cc)
4-13-A1	277.03	108.95	108.89	108.98	108.94	93.90	93.92	93.95	93.92	15.65	16.01	16.08	15.91	162.825	1.701
4-13-B1	255.92	107.20	106.19	105.13	106.17	96.81	97.06	97.50	97.12	16.48	16.15	15.66	16.10	165.987	1.542

Dimensioned: 07/27/01

Hot Vac. Dried: 07/26-27/01

Weighed: 07/27/01

N.B. Ref. No. : 195-129-98

# Impregnant

N.B.R.F.

4-13-A1 195-129-53 (softw by vol to G.P.S+32/Furfural) 195-130-03 ± 04

4-13-B1 " " " " " / " 195-130-05 ± 06

Performed and Recorded by: *J. L. Green*  
Directed by: *J. L. Green*  
Read and Understood by:

Date  
Date  
Date